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Mössbauer study of FINEMET with different permeability

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Abstract Stress field and magnetic field annealed FINEMET ribbons were investigated by ^{57}Fe Mössbauer spectroscopy, magnetic and XRD methods. The change in relative areas of the 2nd and 5th lines in the Mössbauer spectra indicated significant variation in magnetic anisotropy due to the different annealing. High velocity resolution Mössbauer spectroscopy was also used to control the model applied for the evaluation of Mössbauer spectra. A correlation was found between the permeability and the magnetic anisotropy of the annealed FINEMET samples. This can be applied to predict production parameters of FINEMET ribbons with more favorable soft magnetic properties for technological applications.

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1 Introduction

Nanocrystalline $Fe_{73.5}Si_{13.5}Nb_3B_9Cu_1$ FINEMET alloys are widely applied in the industry as excellent soft magnetic material due to high permeability ($\sim 10^5$ at 1 kHz), low magnetostriction ($\sim 2 \times 10^{-6}$), large magnetization ($\sim$1.2 T), low coercivity ($\sim$1 A/m) and low power loss (<5 J/m^3). Production of FINEMET ribbons with more favorable soft magnetic properties for technological applications can be achieved by inducing a transversal magnetic anisotropy via stress annealing [1] or using irradiation with energetic heavy ions [2].

The aim of the present work was to investigate the correlation between the permeability and magnetic anisotropy in annealed nanocrystalline FINEMET samples. For this goal we studied the change in magnetic anisotropy via the change in relative areas of the 2nd and 5th lines in Mössbauer spectra of both stress field and magnetic field annealed FINEMET and checked the phase composition and crystal structure by XRD.

2 Experimental

$Fe_{73.5}Si_{13.5}Nb_3B_9Cu_1$ FINEMET ribbons were rapidly quenched and subsequently stress annealed at 550 °C for 1 h under different tensile stresses between 0 and 124 MPa, obtaining the samples with permeability between 90 and 6000. For comparison, one sample was nanocrystallized at 550 °C/1 h and further annealed under transversal magnetic field of 160 kA/m at 450 °C for 1 h obtaining a permeability as high as 60000.

The ^{57}Fe Mössbauer spectra of the FINEMET samples were taken at 295 K using a conventional Mössbauer spectrometer (WISSEL) in transmission geometry. The magnetic anisotropy was characterized by measuring the relative ratio of the 2nd and 5th lines of sextets, determining the angle θ between the directions of magnetic moment and γ-ray from the formula

$$\frac{A_{2,5}}{A_{1,6}} = \frac{4\sin^2\theta}{3(1+\cos^2\theta)}$$

at the fixed relative ratio of $A_{1,6}/A_{3,4} = 3$.

The γ-rays were provided by a 3×10^9 Bq ^{57}Co/Rh source. Isomer shifts are given relative to α-Fe at room temperature. The Mössbauer spectra were analyzed by least-square fitting of Lorentzian lines by the help of the MOSSWINN code [3].

Selected spectra were also recorded in 4096 channels, with significantly less instrumental error, using an automated precision Mössbauer spectrometric system on the base of the spectrometer SM-2201 with a high velocity resolution at room temperature with moving absorber. Details and characteristics of this equipment and the system were given elsewhere [4–6].

Permeability (μ) of the samples was measured using impedance meter.

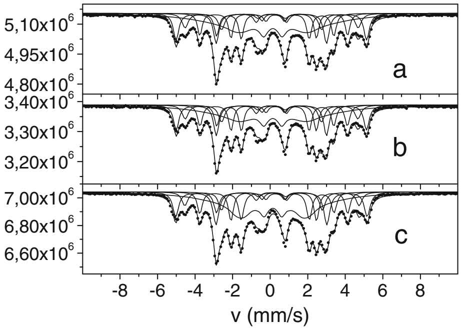

Fig. 1 ^{57}Fe Mössbauer spectra, recorded at $T = 295$ K in 512 channels with conventional velocity resolution, of FINEMET samples with permeability **a** $\mu_r = 170$, **b** $\mu_r = 6000$ and **c** $\mu_r = 60000$

Table 1 Permeability, relative area of 2nd and 5th lines and angle of the average direction of magnetic moment with respect to the normal vector of the ribbon surface of FINEMET samples

Permeability	90	130	170	840	1350	2000	3200	6000	60000
$A_{2,5}/A_{3,4}$	2.85	3.19	3.39	3.70	3.45	3.63	3.59	3.69	4.0
Angle	65.81	70.38	73.34	78.79	74.36	77.32	76.69	78.54	90

Powder X-ray diffractograms of the samples were measured by a computer controlled DRON-2 X-ray diffractometer using $Co_{K\alpha}$ radiation and β filter. The evaluation of the XRD patterns was made by the EXRAY code. For identification of the phases the ASTM X-ray diffraction data were used.

3 Results and discussion

Typical XRD of annealed FINEMET sample reflects nanocrystalline character. The peaks belonging to (2, 2, 0) and (4, 4, 0) reflections of cubic Fe-Si phase with lattice parameter of 0.5669 nm are in good agreement with ASTM card no. 47-1114 and with the earlier results [2].

The Mössbauer spectra measured with a low velocity resolution (Fig. 1) reflect significant changes in the magnetic anisotropy via the changes in the relative areas of the 2nd and 5th lines for the samples having different permeability. We have found gradual change in the average direction of magnetic moment (from 65.8° to 90° with respect to the normal vector of the ribbon surface) with the change of permeability (from $\mu_r = 170$ to $\mu_r = 60000$). The data are shown in Table 1. Figure 2 illustrates the tendency of magnetic anisotropy dependence on the permeability. Our results are consistent with the effect of stress annealing, i.e. that higher stress field applied at annealing induces higher magnetic anisotropy [2]. On the other hand, they also help clarify the controversial data in the literature [1], which were obtained in a relatively narrow permeability interval. The detailed explanation of our findings will be published elsewhere.

The Mössbauer spectra of all samples were decomposed into 5 sextets (3 sextets corresponding to Fe having 4, 5 and 6 nearest Fe neighbors in sublattice A, 1 sextet corresponding to Fe in sublattice B in which Fe has 7 or 8 nearest Fe neighbors in Fe-Si phase and 1 sextet assigned to Fe in amorphous phase) similarly to the case of previous works on the annealed FINEMET [1, 2, 7]. Table 2 shows hyperfine fields of

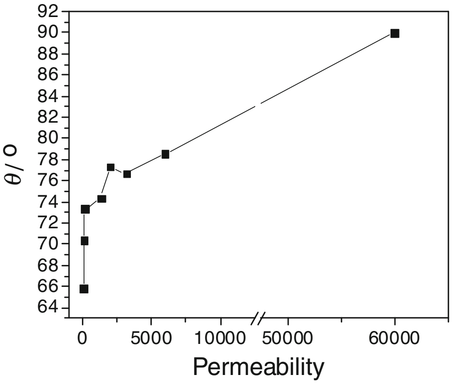

Fig. 2 Dependence of angle of the average direction of magnetic moment on the permeability of FINEMET samples

Table 2 Average hyperfine field parameters of spectral components of FINEMET samples

$A_{2,5}/A_{3,4}$	3.39	3.63	4.0	Fe site
B_1[T]	31.48 (0.01)	31.46 (0.01)	31.63 (0.01)	$D + A_8 + A_7$
B_2[T]	28.58 (0.03)	28.59 (0.02)	28.79 (0.02)	A_6
B_3[T]	24.41 (0.01)	24.39 (0.01)	24.43 (0.01)	A_5
B_4[T]	19.37 (0.01)	19.35 (0.01)	19.42 (0.01)	A_4
B_5[T]	19.01 (0.19)	19.02 (0.13)	18.26 (0.10)	Amorphous

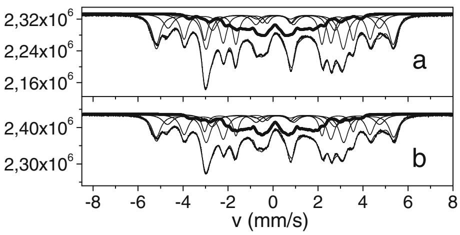

Fig. 3 ^{57}Fe Mössbauer spectra, recorded with a high velocity resolution spectrometer in 4096 channels, of FINEMET samples with permeability **a** $\mu_r = 1350$ and **b** $\mu_r = 6000$. $T = 295$ K

these sextets for selected samples. In all samples the hyperfine fields of the individual components were found to be practically the same. This reflects that, besides the considerable changes found in the magnetic anisotropy, no other significant change in the individual iron microenvironments can be observed at the different stress or field annealing. The reliability of the determination of magnetic anisotropy by the Mössbauer method is enhanced, since the high velocity resolution Mössbauer spectra (Fig. 3) of the FINEMET samples could also be roughly resolved into the 4 sextets of Fe-Si phase, but 3 sextets for the amorphous phase, when the relative areas of the 2nd and 5th lines were in a fairly good agreement with those obtained with the low velocity resolution measurement for the corresponding samples. The detailed analysis of the high velocity resolution spectra will be published in the near future.

4 Conclusion

A new correlation of magnetic anisotropy, obtained from relative areas of the 2nd and 5th lines in Mössbauer spectra, with the permeability has been found in annealed FINEMET samples in a wide range of permeability (from $\mu_r = 170$ to $\mu_r = 60000$). The correlation found can be applied to predict preparation parameters to produce FINEMET ribbons with more favorable soft magnetic properties for technological applications.

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